methylcinnamohydroxamic acid are absent in the other reported compounds.

Registry No. $C_6H_5CH{=}CHCON(CH_3)OH$, 69227-95-8; $C_6H_5CON(OH)C_6H_4$-4-Cl, 1528-82-1; 2-$FC_6H_4CON(OH)C_6H_4$-4-Cl, 94370-35-1; 3-$FC_6H_4CON(OH)C_6H_4$-4-Cl, 94370-36-2; 2,4-$Cl_2C_6H_3CON(OH)C_6H_4$-4-Cl, 94370-37-3; 3,5-$(CH_3O)_2C_6H_3CON(OH)C_6H_4$-4-Cl, 94370-38-4; $C_6H_5(CH_2)_2CON(OH)C_6H_4$-4-Cl, 94370-39-5; $CH_3(CH_2)_4CON(OH)C_6H_4$-4-Cl, 94370-40-8; $CH_3(CH_2)_{14}CON(OH)C_6H_4$-4-Cl, 94370-41-9; 2-$CH_3OC_6H_4CON(CH_3)OH$, 63977-15-1; CH_3NHOH, 593-77-1; 4-ClC_6H_4NHOH, 823-86-9; $C_6H_5CH{=}CHCOCl$, 102-92-1; C_6H_5COCl, 98-88-4; 2-FC_6H_4COCl, 393-52-2; 3-FC_6H_4COCl, 1711-07-5; 2,4-$Cl_2C_6H_3COCl$, 89-75-8; 3,5-$(CH_3O)_2C_6H_3COCl$, 17213-57-9; $C_6H_5(CH_2)_2COCl$, 645-45-4; $CH_3(CH_2)_4COCl$, 142-61-0; $CH_3(CH_2)_{14}COCl$, 112-67-4; 2-$CH_3OC_6H_4COCl$, 21615-34-9; CH_3NO_2, 75-52-5.

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Synthesis and Spectral Characteristics of *N*-Aryl-Substituted Glycines and Alanines

Mohammad Ashique Khadim*† and Lawrence David Colebrook

Department of Chemistry, Concordia University, Montreal, Quebec, Canada H3G 1M8

A series of *N*-aryl ortho-substituted glycines and alanines, which serve as useful intermediates for the synthesis of N^1-aryl-substituted imidazolidine-2,4-diones and their 2-thioxo analogues, have been prepared and characterized by their infrared and carbon-13 and proton NMR spectra. Except for the naphthyl derivatives, all carbon resonances associated with the aryl moieties have been identified by employing relatively simple procedures. Various substituent effects operative in these compounds have been noted. A suitable procedure for the synthesis of *N*-aryl-substituted alanines is described.

Introduction

In connection with our interest in aryl ortho-substituted heterocyclic ring compounds which could exhibit biphenyl-like isomerism (*1-3*), we recently had need of some α-*N*-aryl ortho-substituted glycines and alanines. These amino acids serve as intermediates for the synthesis of N^1-arylimidazolidine-2,4-diones and their 2-thioxo analogues. In this report we describe the synthesis and the carbon-13 and proton NMR and infrared spectra of *N*-aryl ortho-substituted glycines (Ia–h) and alanines (IIa–g). The method of Eckstein et al. (*4*) was found to be a suitable procedure to produce the requisite glycines in adequate yields. However, the procedure described by Miller and Sharp (*5*) for synthesis of *N*-aryl ortho-substituted alanines was found to be unsatisfactory. A suitable procedure for this purpose is described in the Experimental Section.

$$\mathrm{R\text{-}NH\text{-}\overset{2}{C}H(R')\text{-}\overset{1}{C}OOH}$$

I, R′ = H	II, R′ = CH_3
Ia, R = phenyl	IIa, R = phenyl
Ib, R = 2′-tolyl	IIb, R = 2′-tolyl
Ic, R = 2′-methoxyphenyl	IIc, R = 2′-fluorophenyl
Id, R = 2′-fluorophenyl	IId, R = 2′-chlorophenyl
Ie, R = 2′-chlorophenyl	IIe, R = 2′-bromophenyl
If, R = 2′-bromophenyl	IIf, R = 2′,3′-dimethylphenyl
Ig, R = 2′,3′-dimethylphenyl	IIg, R = 1′-naphthyl
Ih, R = 1′-naphthyl	

Except in the case of 1′-naphthyl derivatives, Ih and IIg, all of the carbon-13 resonances have been identified in their NMR spectra. The assignments to various protons and carbon-13 signals in their proton and carbon spectra, respectively, from glycines and alanines were carried out by comparison of their spectra with those of the corresponding primary amines, by estimation of substituent effects (*6*), and, in a few cases, by off-resonance proton decoupling. Various signals of interest in the proton NMR and IR spectra have also been noted. The chemical shift values for given carbon or proton positions in carbon-13 and proton NMR spectra, respectively, for I and II vary over quite a narrow region, and the ranges for different positions of interest are well separated. There is, thus, no ambiguity in the assignment of signals in the carbon-13 and proton NMR spectra. These spectral data should prove very valuable in identification and characterization of compounds of these types.

Carbon-13 chemical shifts for *N*-aryl-substituted glycines, Ia–h, and alanines, IIa–g, are presented in Tables I and II, respectively. Proton NMR spectral data for these compounds are given in Table III. In the case of 2′-fluorophenyl derivatives, Id and IIc, the ^{13}C–^{19}F spin doublets are observable; the magnitude of the coupling constant, *J*, is useful (*7*) in assignment of various carbon signals in these two compounds. NMR signals associated with C-1′ and C-2′ carbons in the 2′-chlorophenyl derivative, Ie, could not be observed, whereas those in the corresponding alanine derivative, IId, showed resonance signals in the expected regions. The carbon signals in the 1′-naphthyl derivatives, Ih and IIg, have only partially been assigned. C-2 carbons experience a significant (6.6 ± 0.3 ppm) but below normal downfield α-effect upon substitution of a hydrogen atom by a methyl group, whereas the downfield β-substitution effect on the carboxyl carbons (C-1) is less pro-

† Present address: Department of Chemistry, University of Virginia, Charlottesville, VA 22901.

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Table I. Carbon-13 NMR Shielding Data for *N*-Aryl-Substituted Glycines (Ia-h)[a]

compd	chemical shift								
	C-1′	C-2′	C-3′	C-4′	C-5′	C-6′	C-2	C-1	aryl ortho
Ia	148.112 (146.8)	112.107 (115.2)	128.745 (129.4)	116.137 (118.7)	128.745 (129.4)	112.107 (115.2)	44.648	172.613	
Ib	145.772 (148.8)	121.610 (121.0)	129.720 (129.4)	116.267 (116.0)	126.730 (125.8)	109.183 (112.0)	44.908	172.678	17.352
Ic	137.324 (133.7)	146.357 (143.5)	109.768 (114.3)	116.202 (117.0)	120.946 (121.0)	109.378 (113.1)	44.583	172.483	55.241
Id	136.544, 136.024,[c] ^2J = 11.76 (135.2)	156.171, 145.708,[c] ^1J = 236.8 (146.9)	114.772, 113.927,[c] ^2J = 19.12 (115.8)	116.267, 116.007,[c] ^3J = 5.89 (117.5)	124.651 (124.2)	112.237, 112.042,[c] ^3J = 4.41 (113.5)	44.323	172.353	
Ie	N[d] (148.5)	N[d] (118.3)	128.810 (129.2)	116.917 (117.4)	127.900 (128.8)	111.393 (113.4)	44.323	172.028	
If	144.343 (151.5)	108.468 (106.6)	132.059 (132.1)	117.567 (117.8)	128.485 (127.1)	111.458 (113.8)	44.583	171.898	
Ig[b]	145.707 (145.2)	119.841 (122.9)	135.699 (138.9)	118.671 (117.4)	125.755 (126.6)	107.558 (107.1)	45.233	172.808	12.413
Ih[e]	143.498 (146.1)	103.139 (112.6)	126.600 (126.9)	C-4′ to C-7′: 116.1-125.6			44.973	172.613	
				(118.3)	(118.3)	(125.9)			

[a] In ppm from Me_4Si, 1.0 M solution in Me_2SO-d_6. Approximate probe temperatures 27 °C. Estimated chemical shifts are given in parentheses; see text. [b] *m*-Methyl at 20.276 ppm. [c] ^{13}C–^{19}F spin doublet. [d] Signal not observed. [e] C-8′, 128.0 (129.1), C-9′, 123.0 (120.4), C-10′, 134.0 (134.7) ppm.

Table II. Carbon-13 NMR Shielding Data for *N*-Aryl Alanines (IIa-g)[a]

compd	chemical shift								
	C-1′	C-2′	C-3′	C-4′	C-5′	C-6′	C-2	C-1	2-Me
IIa	148.093 (140.8)	112.802 (118.8)	129.246 (129.0)	116.703 (123.1)	129.246 (129.0)	112.802 (118.8)	51.388	176.426	18.569
IIb[b]	145.315 (148.8)	122.181 (121.8)	130.006 (130.0)	116.738 (116.6)	126.750 (126.0)	109.982 (112.7)	51.322	175.836	18.419
IIc	135.959, 135.439,[c] ^2J = 11.77 (135.2)	156.106, 145.642,[c] ^1J = 236.8 (146.9)	114.902, 114.057,[c] ^2J = 19.12 (115.8)	116.592, 116.267,[c] ^3J = 7.35 (117.5)	124.521 (124.2)	112.562, ^3J unresolved (113.5)	50.822	175.343	18.002
IId	143.368 (148.5)	117.957 (118.3)	129.655 (129.2)	117.957 (117.4)	128.550 (128.8)	112.367 (113.4)	51.277	175.538	18.717
IIe	143.628 (151.5)	108.988 (106.6)	132.319 (132.1)	118.022 (117.8)	128.680 (127.1)	111.913 (113.8)	50.822	174.888	18.197
IIf[d]	145.187 (145.2)	118.022 (122.9)	135.829 (138.9)	118.931 (117.4)	125.690 (126.6)	108.273 (107.1)	51.537	175.993	18.457
IIg[e]	143.108 (146.1)	103.724 (112.6)	127.900 (126.9)	C-4′ to C-9′: 116.3-126.5			51.472	175.863	18.002
				(118.3)	(118.3)	(125.9)			

[a] In ppm from Me_4Si, 1.0 M solution in Me_2SO-d_6. Approximate probe temperatures 27 °C. Estimated chemical shifts are given in parentheses; see text. [b] *o*-Methyl at 17.447 ppm. [c] ^{13}C–^{19}F spin doublet. [d] *o*-Methyl at 12.543 ppm; *m*-methyl at 20.341 ppm. [e] C-10′, 134.0 (134.7) ppm.

Table III. Proton NMR Chemical Shifts[a] of *N*-Aryl-Substituted Glycines (Ia-h) and Alanines (IIa-g)

	chemical shift					
compd	CH_2[s]	CH[q]	CH_3[d]	$ArCH_3$[s]	OH[s] and NH[s,b]	aryl[m]
Ia	4.00				N	6.50–7.0
Ib	3.97			2.17 (2′)	8.33–9.33	6.50–7.33
Ic[t]	4.00				8.60–9.60	6.53–7.20
Id	4.00				N	6.67–7.50
Ie	4.07				N	6.67–7.77
If	4.03				N	6.57–7.77
Ig	4.00			2.13 (2′)	8.67–9.33	6.37–7.33
				2.32 (3′)	9.33–10.17	6.57–8.57
Ih	4.23					
IIa		4.08	1.42		9.0–9.7	6.57–7.50
IIb		4.17	1.50	2.15 (2′)	8.40–9.17	6.60–7.40
IIc		4.04	1.49		N	6.7–7.5
IId		4.30	1.48		N	6.7–7.6
IIe		4.31	1.50		N	6.7–7.8
IIf		4.12	1.47	2.10 (2′)	8.4–9.1	6.5–7.2
				2.27 (3′)		
IIg		4.26	1.62		N	6.5–8.7

[a] In ppm from internal Me_4Si; spectra determined at 60 MHz, 1.0 M solutions in Me_2SO-d_6. b = broad signal with no fine structure; m = multiplet; d = doublet; q = quartet; s = singlet; t = 2′-OCH_3, 3.93 ppm, s; N = not observed.

nounced (3.5 ± 0.2 ppm; Table IV). A small upfield γ-effect (0.0–0.7 ppm) on C-1′ aryl carbons is observed arising from the C-2 methyl substituent. Upon incorporation of a glycine or alanine moiety into the framework of imidazolidine-2,4-diones (or 2-thioxo-4-imidazolidinones), the magnitude of various substituent effects does not change to any considerable extent (*3*). The δ effects of aryl ortho substituents on C-2 carbons have a slight but noticeable dependence on the nature of aryl ortho substituents. The electron-releasing substituents show small but consistent deshielding δ-shifts on C-2 of the order of 0.0–0.6 ppm, whereas in derivatives with electronegative substituents, Ic–f and IId–f, the δ-shifts are consistently upfield (0.0–0.6 ppm). The δ-methyl shifts on C-6′ are consistently downfield and relatively large (0.4–1.0 ppm), and the effect on the C-2′ ortho carbons is variable; in Ig in particular, a large upfield effect of 1.8 ppm is observed. Since the *m*-methyl group is aryl systems is known to exert a small downfield (~0.8 ppm) β-effect, and a considerable upfield (~3 ppm) γ-effect (*5*), the sign and magnitude of δ-methyl effects on aryl ortho C-2′ and C-6′ carbons are unexpected.

Experimental Section

The broad-band decoupled, pulsed Fourier transform carbon-13 NMR spectra were measured in Me_2SO-d_6 (1.0 M solutions) by using a Brucker WH-90 NMR spectrometer operating at 22.63 MHz, with a probe temperature of 27 °C. The solvent provided the heteronuclear deuterium lock signal. Pulse widths of 5 μs, a repetition time of 0.6–0.7 s, a spectral width of 6024 Hz, and 2000–5000 scans were typically employed. Chemical shifts are reported relative to Me_4Si. Proton NMR spectra were measured with a Varian A-60 NMR spectrometer at 60 MHz; 1.0 M solutions were prepared in Me_2SO-d_6.

N-Aryl-substituted glycines were synthesized following the method of Eckstein (*4*) et al. In our laboratories, the procedure described by Miller and Sharp (*5*) for the preparation of *N*-aryl-substituted alanines was found to be unsatisfactory. The yields obtained were low (10–20%). A suitable procedure for preparation of *N*-aryl-substituted alanines is given below.

A mixture of aromatic primary amine (0.10 mol), sodium acetate trihydrate (0.10 mol), 3–4 mL of ethanol, and ethyl 2-bromopropionate (0.10 mol) was stirred and heated to reflux for 20–24 h. After being cooled to room temperature and diluted with 40 mL of water, the mixture was extracted with ether (3 × 50 mL) and the combined extracts were evaporated on a rotary evaporator. The oily residue was then heated to reflux with 70–80 mL of 10% aqueous NaOH for 2 h, and the mixture was cooled to room temperature, and washed with ether (3 × 25 mL). The aqueous layer was cooled in an ice–water mixture, and the solution was acidified with slow addition of concentrated HCl until the pH was 2–3. The resulting suspension (or slurry in some cases) was cooled in an ice–water mixture for 2–3 h, filtered, washed successively with some water and a large quantity of hexane, and dried under high vacuum. Finally, the precipitate was recrystallized from chloroform, washed with hexane, and again dried under high vacuum overnight. Percent yields and melting points are given in Table IV. All glycines (Ia–h) and alanines (IIa–g) were characterized by C, H, and N elemental analysis, and by carbon-13 and proton NMR and IR spectra. Infrared spectra showed the following characteristic absorptions (KBr, cm^{-1}): glycines Ia–h, NH (3470–3500, m), C=O (1725–1750, s), and OH (3200–2400, b); alanines IIb–g, NH (3410–3500, m), C=O (1720–1750, s), and OH (3200–2500, b); IIa, NH (3450, m), C=O (1600, s), and OH (3000–2600, b). m, s, and b represent medium, strong, and broad signals, respectively. Elemental analysis for a sample compound, Ic, was found to be C, 59.5; H, 6.3; N, 7.9; calculated C, 59.7; H, 6.1; N, 7.7. The purity of the products was also checked by thin-layer chromatography. These compounds were derivatized and further characterized by their reaction with phenyl isothiocyanate in order to synthesize 1,3-diaryl-2-thioxo-4-imidazolidinones for which elemental analysis was found to be satisfactory (*8*). For example, elemental analysis of a derivative of Ic, 3-phenyl-1-(2-methoxyphenyl)-2-thioxo-4-imidazolidinone, $C_{16}H_{14}N_2O_2S$, was found to be C, 64.5; H, 5.1; N, 10.1; calculated C, 64.4; H, 4.7; N, 9.6. The latter series of compounds was also characterized by carbon-13 and proton NMR and IR spectra (*3*, *8*).

Table IV. Carbon-13 Methyl Group Substituent Shifts[a] (ppm), Melting Points,[b] and Experimental Yields of *N*-Aryl-Substituted Glycines (Ia–h) and Alanines (IIa–g)

	α-shifts	β-shifts	γ-shifts	δ-shifts on							
compd	on C-2	on C-1	on C-1′	C-2′	C-6′	glycine	mp, °C	yield, %	alanine	mp, °C	yield, %
Ia, IIa	6.74	3.81	−0.02	0.70	0.70	Ib	147.0–148.0	24	IIa	159.0–160.0	70
Ib, IIb	6.41	3.16	−0.46	0.57	0.80		148 (*9*)		IIb	111.0–111.5	58
Id, IIc	6.50	3.00	−0.59	−0.07	0.42	Ic	148.0–149.0	59	IIc	122.5–123.0	56
Ie, IId	6.95	3.51	N[c]	N[c]	0.97	Id	120.0–121.0	17	IId	147.5–148.0	52
If, IIe	6.24	3.00	−0.72	0.52	0.45	Ie	167.0–168.0	18	IIe	163.5–164.5	47
Ig, IIf	6.30	3.18	−0.52	−1.82	0.71		171 (*5*), 169 (*9*)		IIf	124.0–125.0	60
Ih, IIg	6.50	3.25	−0.39	0.59	r[d]	If	165.5–166.5	31	Ig	146.0–147.0	82
						Ig	150.0–151.0	32			
						Ih	187.0–188.0	56			

[a] Shifts measured relative to the less substituted compound. A negative number represents a displacement in the shielding direction. [b] All melting points are uncorrected. [c] C-1′ and C-2′ resonances not observed. [d] Resonance not identified.

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We thank the National Research Council of Canada for grants to L.D.C., and Drs. A. S. Perlin and G. K. Hamer, Pulp and Paper Institute of Canada, Montreal, Canada, for their help with some of the spectra.

Registry No. Ia, 103-01-5; Ib, 21911-61-5; Ic, 94800-23-4; Id, 5319-42-6; Ie, 6961-49-5; If, 40789-38-6; Ig, 83442-59-5; Ih, 6262-34-6; IIa, 15727-49-8; IIb, 94800-25-6; IIc, 94800-27-8; IId, 94800-29-0; IIe, 94800-31-4; IIf, 94800-33-6; IIg, 94943-86-9; phenylamine, 62-53-3; 2-tolylamine, 95-53-4; 2-fluorophenylamine, 348-54-9; 2-chlorophenylamine, 95-51-2; 2-bromophenylamine, 615-36-1; 2,3-dimethylphenylamine, 87-59-2; 1-naphthylamine, 134-32-7; ethyl 2-bromopropionate, 535-11-5.

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Corrections

Conductances, Transference Numbers, and Activity Coefficients of Some Aqueous Terbium Halides at 25 °C. Frank H. Spedding,* Robert A. Nelson, and Joseph A. Rard, *J. Chem. Eng. Data* **1974**, *19*, 379–81.

In eq 4, the first E should appear immediately after the curly brace, not before. That is

$$\ln\left(\frac{y_{\pm}}{y_{\pm}'}\right) = \ln\left(\frac{c'}{c}\right) - \frac{3F}{4RT}\left\{\frac{E}{t_+'} + \int_0^E \left(\frac{1}{t_+} - \frac{1}{t_+'}\right) dE\right\}$$

The calculations were done correctly.

Relative Viscosities of Some Aqueous Rare Earth Perchlorate Solutions at 25 °C. Frank H. Spedding,* Loren E. Shiers, and Joseph A. Rard, *J. Chem. Eng. Data* **1975**, *20*, 66–72.

In Table I, seventh from the last entry, the correct molality is 2.4016 and not 1.4016.

A Review of the Osmotic Coefficients of Aqueous H_2SO_4 at 25 °C. Joseph A. Rard, Anton Habenschuss, and Frank H. Spedding,* *J. Chem. Eng. Data* **1976**, *21*, 374–9.

On p 375, five lines above eq 2, the value of B should be −1194 cm^3/mol and not −1994 cm^3/mol. The correct value was used in calculations. Also, the first entry in Table I for Scatchard, Hamer, and Wood—Isopiestic vs. NaCl should have $\phi = 0.6776$, not 0.6676.

Heats of Dilution of Some Aqueous Rare Earth Electrolyte Solutions at 25 °C. 2. Rare Earth Nitrates. Frank H. Spedding,* John L. Derer, Michael A. Mohs, and Joseph A. Rard, *J. Chem. Eng. Data* **1976**, *21*, 474–88.

In Table I, the data for Terbium Nitrate are mislabeled as Dysprosium Nitrate and vice versa (the least-squares parameters and graphs are correct). Also, on p 484 (first column, last line), it should read "$-\bar{L}\cdot$ is the heat of solution to form an infinitely dilute solution...". That is, $\bar{L}\cdot$ in Table II are the negatives of the heats of solution.

Isopiestic Determination of the Osmotic Coefficients of Aqueous Na_2SO_4, $MgSO_4$, and Na_2SO_4–$MgSO_4$ at 25 °C. Joseph A. Rard* and Donald G. Miller, *J. Chem. Eng. Data* **1981**, *26*, 33–8.

On p 36, fifth line from bottom in first column, the molality should read 0.075 mol kg^{-1} and not 0.025 mol kg^{-1}.

Densities and Apparent Molal Volumes of Aqueous Manganese, Cadmium, and Zinc Chlorides at 25 °C. Joseph A. Rard* and Donald G. Miller, *J. Chem. Eng. Data* **1984**, *29*, 151–6.

In eq 1, the exponent of X is $I/2$, not 1/2.